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X-RAY DAMAGE TO CF_3CO_2 -TERMINATED ORGANIC MONOLAYERS ON Si/Au SUPPORTS
IS DUE PRIMARILY TO X-RAY INDUCED ELECTRONS

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Technical Report No. 44 (December 1991)

Interim Technical Report
(Accepted for publication in Science)

PREPARED FOR DEFENSE ADVANCED RESEARCH PROJECTS AGENCY
1400 Wilson Boulevard
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DEPARTMENT OF THE NAVY
Office of Naval Research, Code 1130P
800 North Quincy Street
Arlington VA 22217-5000

Project No.: a400011dd205
Contract No.: N00014-86-K-0756
Effective Date: 86 September 15
Expiration Date: 92 September 14

91-18206



Principal Investigator: George M. Whitesides
(617) 495-9430

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) Technical Report No. 44			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
6a. NAME OF PERFORMING ORGANIZATION Harvard University	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION DARPA			
6c. ADDRESS (City, State, and ZIP Code) Office for Sponsored Research Holyoke Center, Fourth Floor Cambridge MA 02138-4993		7b. ADDRESS (City, State, and ZIP Code) 1400 Wilson Boulevard Arlington VA 22209-2308			
8a. NAME OF FUNDING/SPONSORING ORGANIZATION ONR	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER			
8c. ADDRESS (City, State, and ZIP Code) Chemistry Division, Code 1113 Office of Naval Research Arlington VA 22217-5000		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO. 86-K-0756	PROJECT NO.	TASK NO. a400011dd2	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) "X-ray Damage to CF ₃ CO ₂ -Terminated Organic Monolayers on Si/Au Supports is due Primarily to X-ray Induced Electrons."					
12. PERSONAL AUTHOR(S) P.E. Laibinis, R.L. Graham, H.A. Biebuyck, and G.M. Whitesides					
13a. TYPE OF REPORT Technical	13b. TIME COVERED FROM _____ TO _____	14. DATE OF REPORT (Year, Month, Day) December, 1991		15. PAGE COUNT	
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP			
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
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20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL Dr. Harold Guard			22b. TELEPHONE (Include Area Code) 703/696-4311		22c. OFFICE SYMBOL

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**X-ray Damage to CF₃CO₂-terminated Organic Monolayers on Si/Au
Supports is Due Primarily to X-ray Induced Electrons.**

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DTIC TAB	SI
Unannounced	SI
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Availability Codes	
Dist	Avail and/or Special
A-1	

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Abstract

X-rays damage organic materials. The relative importance of the X-rays themselves, and of both X-ray-generated and secondary electrons, in this damage was explored using self-assembled monolayers (SAMs) on multilayer thin-film supports. The substrates were prepared by depositing thin films of Si (0, 50, 100 and 200 Å) on thick layers of Au (2000 Å); these systems were supported on chromium-primed silicon wafers. Trifluoroacetyl-terminated SAMs were assembled on these substrates and the samples irradiated with monochromatic Al K α X-rays. The fluxes of X-rays to which the different samples were exposed were the same, but the fluxes and energy distributions of the electrons generated by interactions of the X-rays with the substrates differed. The loss of fluorine from the SAMs was followed by XPS and was slower on substrates emitting a lower flux of electrons. This observation indicated that the electrons, and not the X-rays themselves, were largely responsible for the damage to the organic monolayer films that resulted in loss of fluorine from them.

X-rays damage organic materials (1). This damage can either limit their utility or provide the basis for useful technologies (e.g. X-ray lithography (2,3) or radiation cross-linking of polymers (3)). Understanding the mechanisms of the damage accompanying exposure to X-rays is helpful in designing materials and environments in which this exposure yields the desired results. A basic tenet of areas of technology involving X-ray processing has been that X-rays do not interact strongly with matter (4) and should effect little damage to materials; the photo-generated primary and secondary electrons interact more strongly, and have been postulated to be the damaging species (5).

We and others (6) have recently begun to explore the mechanisms of X-ray-induced damage to organic materials, employing as samples self-assembled monolayers (SAMs) on metal substrates (7-9). SAMs are well suited for this study because they allow a variety of organic functionalities to be incorporated into monomolecular films having well-defined structures. SAMs can be easily generated having dimensions of interest (10-40 Å; the characteristic escape depth (10) for electrons in the energy range encountered -- ≤ 1.5 keV -- is 20-40 Å). A range of substrates can be derivatized using different types of SAMs: two we employ here are alkanethiolates on gold (7), and alkylsiloxanes on silica.^{9,10} We have chosen the trifluoroacetyl group (CF_3CO_2^-) as a probe for a number of reasons: its surface concentration is easily measured by XPS (11); it decomposes rapidly when irradiated with X-rays (6); it is easily introduced into SAMs (7,8); it is localized at the monolayer-air interface and analysis of the concentration of fluorine in the SAM is not complicated by the presence or generation of other signals.

In this report, we outline a study of the rate of damage to trifluoroacetyl groups attached via an undecyl tether ($\text{CF}_3\text{CO}_2(\text{CH}_2)_{11}-$) to the surface of thin (50-200 Å) silicon films supported on gold. The objective of this study was to examine the

relative rates of damage to the organic components of the system -- the CF_3CO_2 -terminated SAM -- as a function of intensities of X-ray photons and of electrons. The results provide direct experimental confirmation that, under conditions relevant to technologies such as X-ray photolithography, electrons are responsible for the majority of damage in one representative organic system. The electrons measured in this study include primary (photo- and Auger) and secondary electrons; we refer to these collectively as electrons.

To vary the number of electrons generated by interaction of the X-rays with the support, we prepared composite substrates (Figure 1) comprising materials -- gold and silicon -- characterized by very different electron yields on X-ray irradiation; gold produces a higher electron yield than silicon (11). To generate intensities of electrons at the CF_3CO_2 group covering a range of values, we coated thick films of gold (~ 2000 Å) with various thicknesses of evaporated silicon (50, 100 and 200 Å as determined *in situ* using a quartz crystal microbalance; each $\pm 10\%$). X-rays are not significantly attenuated by these thicknesses of silicon (4), and the number of X-ray photons in the gold films -- and thus the number of X-ray induced photoelectrons from the gold -- is similar in all of these samples. The electrons generated in the gold are, however, strongly scattered by the silicon.

To confirm the structure of the substrates, we used Rutherford Backscattering (RBS) (12) employing 3 MeV He^{+2} for independent characterization of the thickness of the silicon overlayer. Figure 2 shows representative spectra; the signals associated with silicon (inset) were analyzed (13) and thicknesses of 50, 105, and 185 Å (values estimated to be ± 10 Å) were inferred for the three samples. The width of the peak associated with gold suggests the reproducibility of the thicknesses of the multilayer substrates we fabricate ($\sim \pm 10\%$). The spectra confirm that neither chromium nor bulk silicon are localized at the substrate/vacuum interface; RBS lacked the sensitivity to determine whether gold

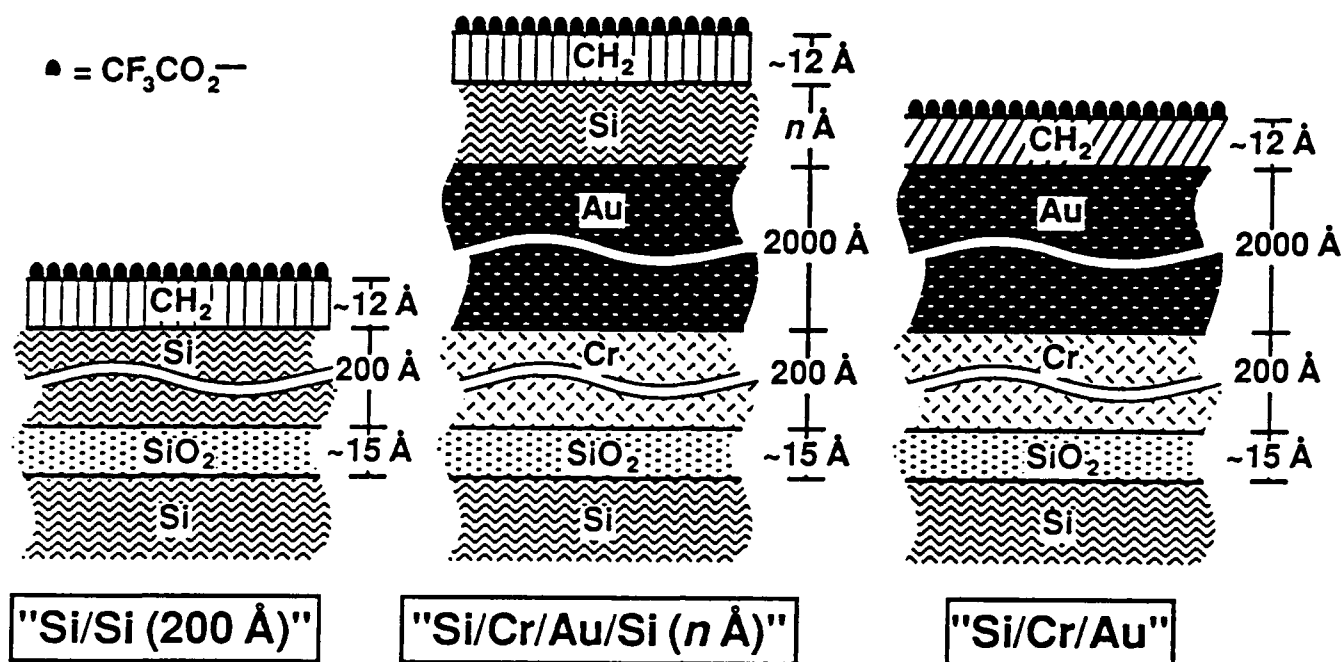
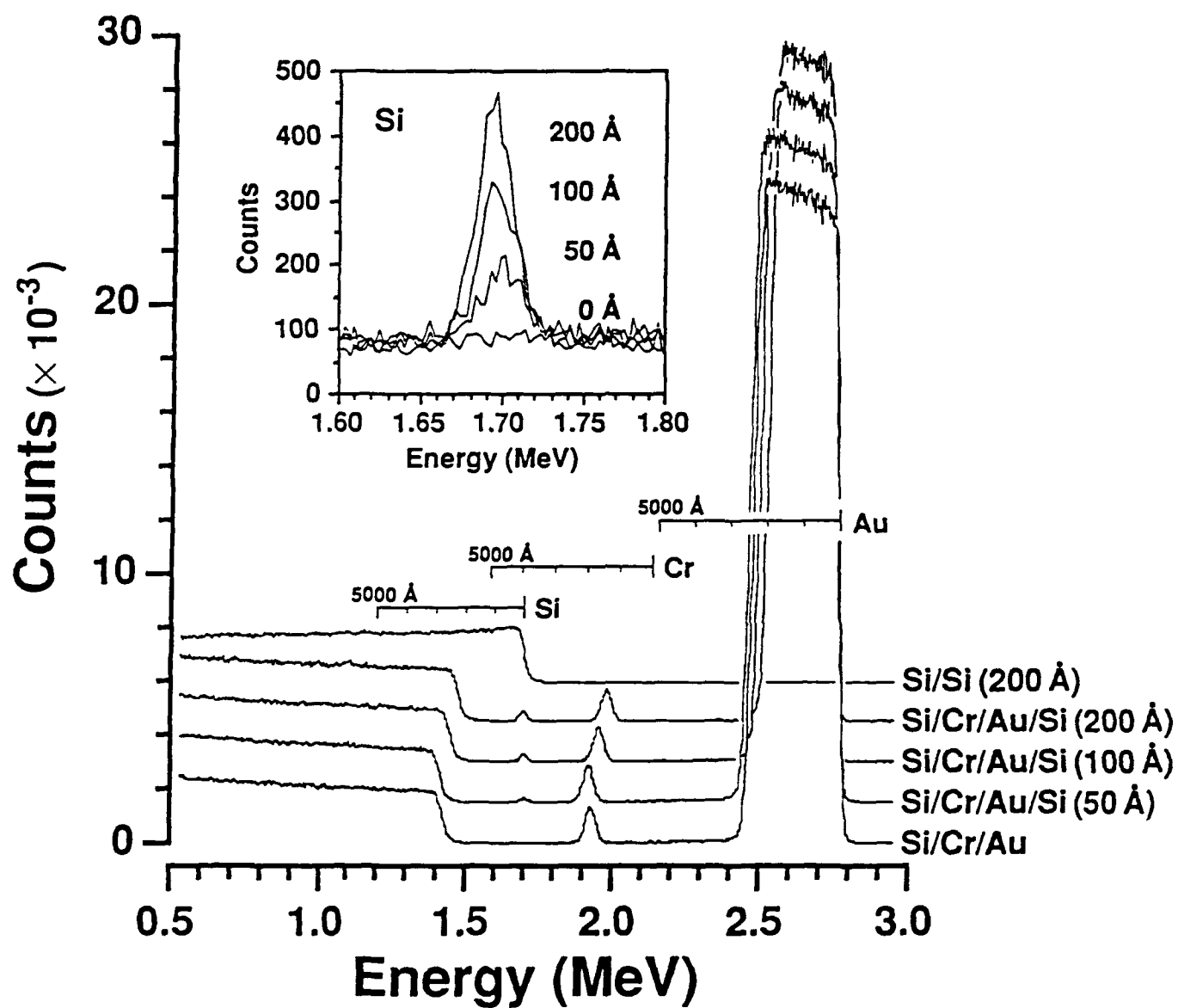


Figure 1. Schematic illustration of the structures employed in these studies. The lines denoting interfaces between materials are not meant to indicate that the interfaces are atomically flat; we have not measured their flatness. The lowest layers of Si represent Si(100) wafers (~0.5 mm thick) on which the assemblies were supported; the other layers of Si were prepared by evaporation and are probably amorphous. The evaporated films of Si contain a layer of SiO₂ (not shown) of undetermined thickness (but probably ~15 Å). Chromium was employed as an adhesion layer between the Si wafer and gold.

Figure 2. Rutherford backscattering (RBS) spectra of Si/Cr/Au/Si substrates for different thicknesses of the silicon overlayer. The substrates were prepared by the sequential evaporation onto the surface of a silicon wafer (Si(100) with ~ 15 Å of native oxide) of 200 Å of Cr, 2000 Å of Au and n Å ($n = 0, 50, 100, 200$) of Si. The thicknesses were determined using a quartz crystal microbalance (QCM) in the evaporator chamber (± 10 %). Quantitation of the signals associated with the silicon layer (inset) yielded values of its thickness (error estimated to be ± 10 Å): QCM (RBS) 50 Å (50 Å); 100 Å (105 Å); 200 Å (185 Å). The spectrum of silicon is provided for comparison. The markers each illustrate the location of a particular element relative to the vacuum interface; for example, Cr is located ~ 2000 Å beneath the surface. The assignment of signals is (eV) Au: 2.4-2.8; Cr: 1.9-2.0; Si: 1.7, and the plateaus for $E \leq 1.7$. The spectra are displaced vertically for clarity.



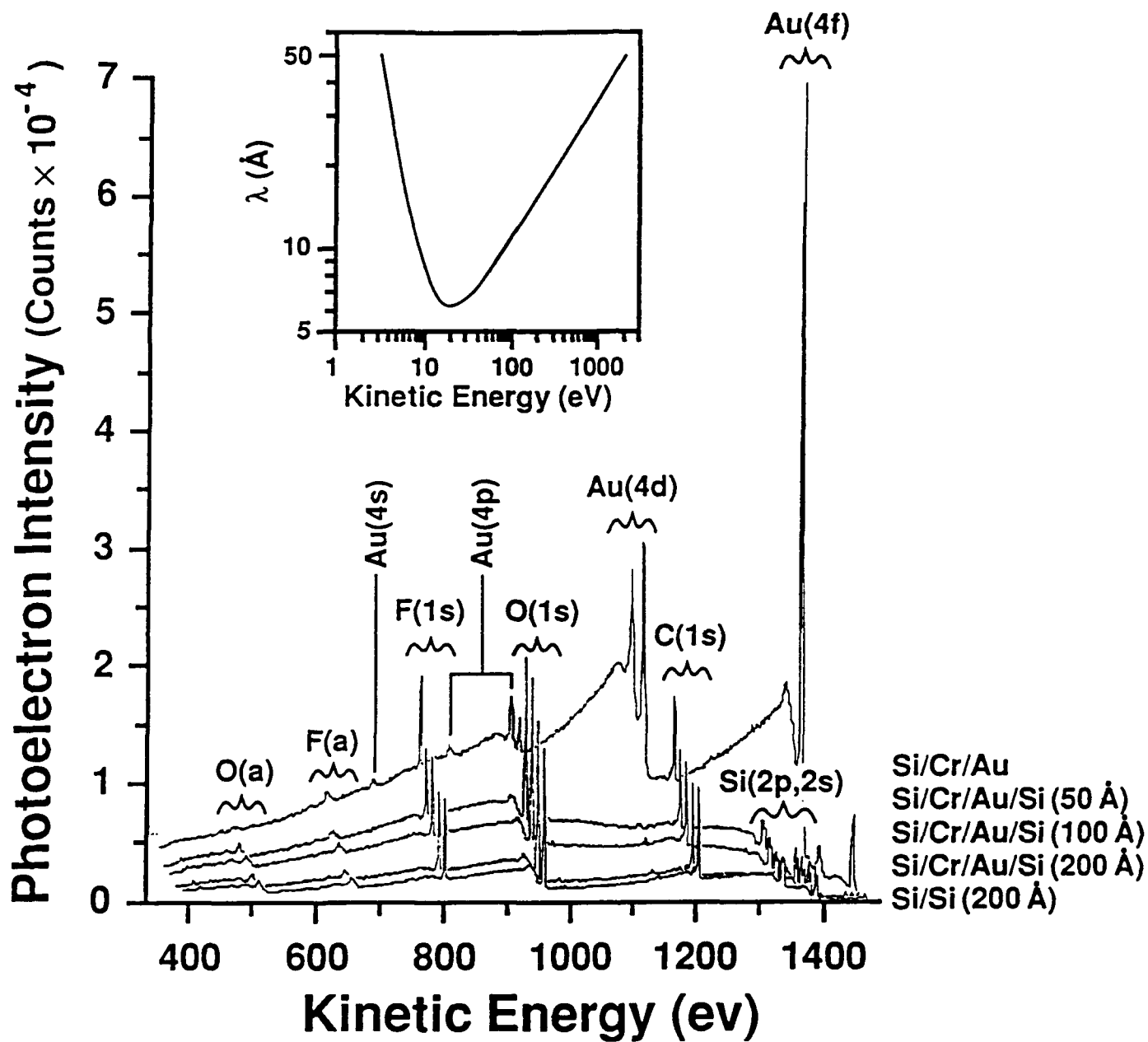
or silicon was present at the surface. By X-ray photoelectron spectroscopy (XPS), the films coated with silicon exhibited no peaks due to gold (only silicon, oxygen and carbon) and were found to be unreactive to alkanethiols (14). These observations suggest that the silicon layers are essentially free of pinholes.

The surfaces were derivatized by the self-assembly technique (7-9). SAMs were formed on the substrates exposing SiO_2 by allowing them to react with $\text{CH}_2=\text{CH}(\text{CH}_2)_9\text{SiCl}_3$ in hexadecane; the resulting olefin surfaces were transformed into trifluoroacetate surfaces via a two-step procedure using $\text{BH}_3/\text{H}_2\text{O}_2$ and trifluoroacetic anhydride (TFAA) (8). SAMs were formed on gold by reaction with $\text{HS}(\text{CH}_2)_{11}\text{OH}$ in deoxygenated absolute ethanol; the alcohol surface was converted to the trifluoroacetate by 30 s exposure to 5% TFAA/hexanes.

Figure 3 displays XPS spectra of representative derivatized substrates. The figure demonstrates that the electron flux through the SAMs decreased as the thickness of the silicon overlayer increased. While peaks directly attributable to gold were not observed when the silicon overlayer was $\geq 50 \text{ \AA}$, we believe that the photoelectrons due to gold were, after inelastic collisions, largely responsible for the increased baseline (relative to pure silicon) for the samples containing an overlayer of silicon. A 200- \AA overlayer of silicon virtually masked the presence of the underlying gold: the spectra of this system and that having only silicon as substrate are very similar. Figure 3 further establishes that the intensity of the $\text{F}(1s)$ peak from the CF_3CO_2 groups of the SAMs on the various substrates were approximately equal: the different methods of forming SAMs generated approximately equal numbers of CF_3CO_2 groups per unit area of surface (15).

The samples were exposed to a constant flux of monochromatized Al K_α X-rays ($E = 1486.6 \text{ eV}$, anode power = 200 watts) in a Surface Science SSX-100 XPS spectrometer (operating pressure = 10^{-9} torr) using a spot of size $\sim 1 \text{ mm}^2$.

Figure 3. XPS spectra of trifluoroacetyl-terminated monolayers on Si/Cr/Au/Si substrates. The spectra were obtained on spots that had been previously unexposed to X-rays and required ~15 min of exposure to the beam; the amount of damage to the SAM during this exposure is small (<5 %). The spectra have been offset horizontally by -10 eV from the one below it for clarity; *the spectra have not been offset vertically*. There is no residual gold signal even in the thinnest Si film (50 Å), and thus, we believe, no pinholes. This conclusion is reinforced by other experiments reported in the text. The hydroboration procedure incorporated a contaminant, Ca (KE ~ 1100 eV), that is present in <1 atomic %. As we compare the rate of damage to electron intensity, presence of an impurity of Ca is not important. Inset: The "universal" curve (10). λ , the inelastic mean free path, is defined as the distance at which the probability an electron transversing a medium without significant energy loss is $1/e$. $1/\lambda$ is directly related to the probability of an electron interacting inelastically with a medium.



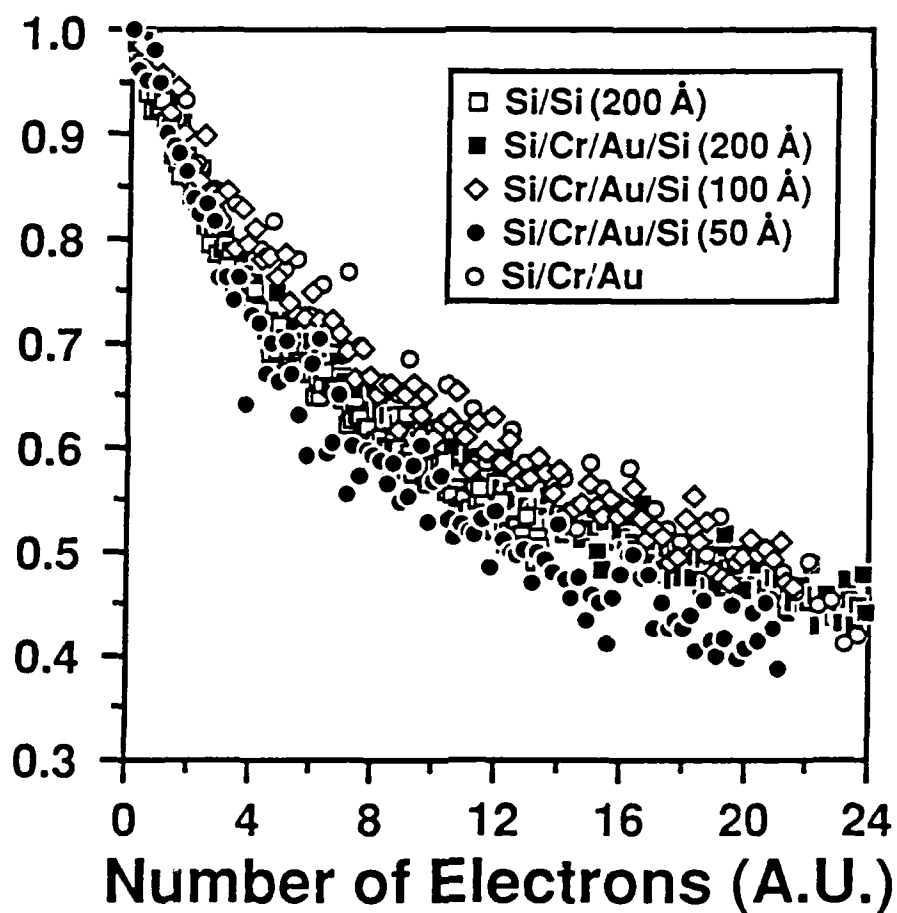
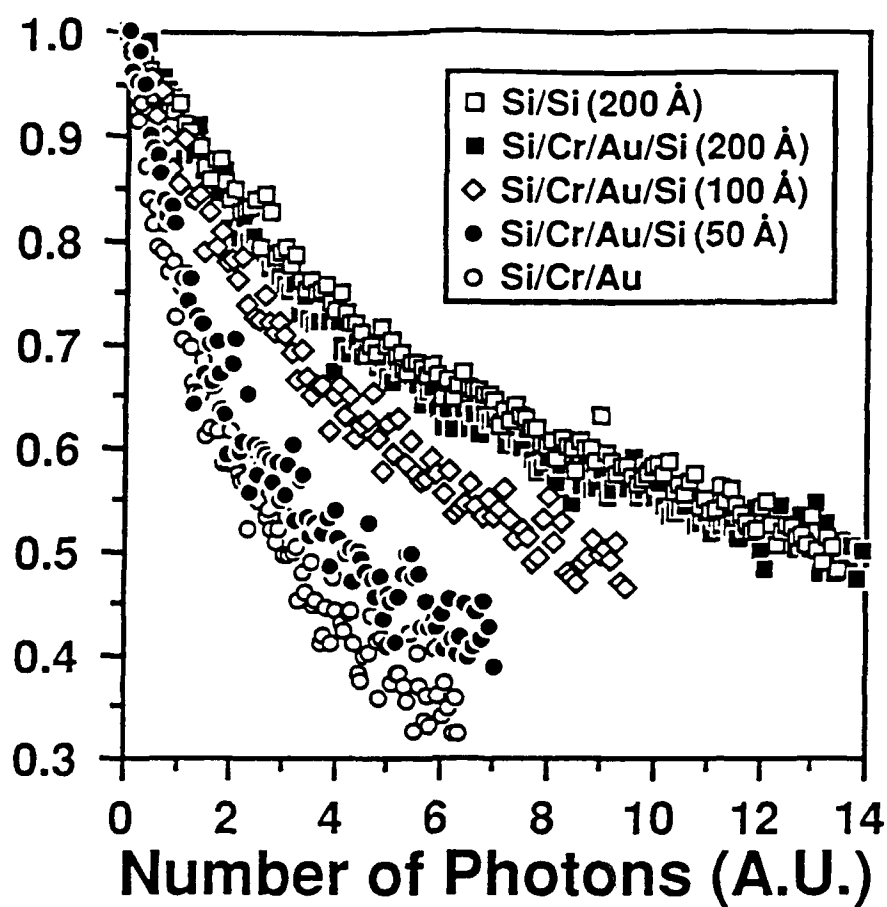
Electrons due to F(1s) were detected using a concentric hemispherical analyzer (pass energy = 100 eV).

Figure 4 summarizes the relative intensity of the F(1s) peak upon exposure of the CF₃CO₂-terminated monolayers on the various substrates to Al K_α X-rays. The intensity of the peak was measured during sequential 4 minute intervals. With continued exposure to X-rays, the intensity of the F(1s) peak decreased. The amount of loss of fluorine from the various substrates *exposed to common numbers of photons* is different (upper panel); the loss of fluorine is faster on the substrates that exhibit greater intensities of electrons upon irradiation with X-rays (Figure 3). The different amounts of damage that occur upon exposing identical monolayers on substrates with different electron yields to a common intensity of X-rays demonstrate that electrons are, at some level, important in causing X-ray-induced damage.

The relative importance of photons and electrons in causing damage to the SAMs can be estimated using the data in Figure 3. The flux of electrons from the various substrates under a common flux of photons is different. We have estimated the relative intensities of electrons from the substrates by integrating (16,17) the XPS spectra in Figure 3 over the energy range kinetic energy (KE) = 387 to 1487 eV. We are limited by the electrostatic analyzer on the XPS to quantitation of electrons of KE \geq 400 eV and we acknowledge that there may be systematic errors in estimating the relative intensities of electrons in the range of energies that are most damaging (~50 eV) from the observed yield of electrons of energies > 400 eV (17,18). In Figure 4 (lower panel), we plot the intensity of F(1s) from the various substrates vs. the number of electrons to which the SAM has been exposed. To generate similar doses of electrons, the samples containing thicker overlayers of silicon were exposed to the X-ray beam longer than those having thinner overlayers. The profiles in Figure 4 (lower panel) are remarkably

Figure 4. X-ray induced damage to trifluoroacetyl-terminated monolayers on Si/Cr/Au/Si substrates as a function of the number of photons (upper) and electrons (lower) to which the CF_3CO_2^- group were exposed. The intensity of the F(1s) signal was determined from sequential scans (~4 min each). The number of electrons from these samples were estimated by integrating (16,17) the XPS spectra in Figure 3. In the lower panel, the substrates require different lengths of exposure to the X-ray beam to yield similar numbers of electrons. The different rates of loss evidenced in the upper panel and the similarity of the profiles in the lower panel suggest that the damage to the SAMs results primarily from the electrons and not from the photons themselves. A.U. = arbitrary units.

F(1s) Normalized Intensity



similar (especially given that the substrates had different lengths of exposure to the X-ray beam) and suggest that the primary and secondary electrons are much more important in the damage process than are the X-ray photons. The deviations present are, we believe, primarily due to difficulties in maintaining a constant photon flux.

While the present data do not determine whether electrons are *solely* responsible for causing damage, they are, however, consistent with primary and secondary electrons being responsible for most (and maybe all) of the damage to a representative organic system upon irradiation with X-rays.

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4. For example, the penetration depth of X-rays (KE ~2 kV) into organic materials is of the order of microns; of electrons, it is of the order of ~100 Å [M. P. Seah, in *Practical Surface Analysis*, D. Briggs and M. P. Seah, Eds. (John Wiley & Sons, Chichester, 1983), Chapter 5]. In our experiment, we estimate the flux of X-ray photons experienced by the CF₃CO₂- group to be more than ~100 times greater than the flux of electrons.
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10. The so-called "universal" curve (Figure 3, inset) relates the inelastic mean free path of an electron, λ , (the distance required to reduce the probability an electron escaping to 1/e) through a material to the kinetic energy of the electron [M. P. Seah and W. A. Dench, *Surf. Interface Anal.* **1**, 2 (1979)].
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13. RBS spectra were analyzed using "SA" (Spectrum Analysis), a PC version of the TEK program for RBS analysis developed at Oak Ridge. SA was written by Patrick M. Smith (Harvard University, Division of Applied Sciences).

14. To test for the presence of gold at the surface of the Si/Cr/Au/Si substrates, the Si-coated gold substrates were immersed in an ethanolic solution containing a fluorine-containing alkanethiol ($\text{CF}_3\text{CF}_2\text{CH}_2\text{O}(\text{CH}_2)_{11}\text{SH}$) overnight. Thiols react with soft metal/metal oxides -- for example, gold -- but do not react with hard metal/metal oxides -- alumina, silica [P. E. Laibinis, J. J. Hickman, M. S. Wrighton, G. M. Whitesides, *Science* **245**, 845 (1989); P. E. Laibinis and G. M. Whitesides, unpublished results]. No fluorine was detected on any substrate exposing Si by XPS suggesting that the Si-coated gold substrates reveal no (or very little) gold at their surface. We estimate that we would have detected 0.1 % of a monolayer of the fluorinated thiolate.

15. We have estimated that the procedure used to derivatize silicon yields ~80% the number of trifluoroacetyl groups as that used to derivatize gold (8).

16. In estimating the relative flux of electrons from the various substrates, the spectra (Figure 3) were corrected for differences in the efficiency of the electrostatic analyzer with the KE of the electron. The effect of this correction on the relative electron flux is small (<2 %).

17. We have also analyzed the XPS spectra incorporating differences in the likelihood of an electron to interact with the SAM (estimated as $1/\lambda$) and find that the correction would be small (≤ 3 %); the direction of this correction is opposite that due to detector inefficiencies (16).

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19. This research was supported in part by the Office of Naval Research, the Defense Advanced Research Projects Agency, and the National Science Foundation (Grant CHE-88-12709). XPS and RBS spectra were obtained using instrumental facilities purchased under the DARPA/URI program and maintained by the Harvard University Materials Research Laboratory. We thank John F. Chervinsky and Patrick M. Smith (Harvard University, Division of Applied Sciences) for help in characterizing the substrates by RBS, John P. Folkers for supplying the $\text{H}_2\text{C}=\text{CH}(\text{CH}_2)_9\text{SiCl}_3$, Dr. Colin D. Bain (Cambridge University) for invaluable discussions and suggestions, and our colleagues Steven M. Bonser, John P. Folkers, and John P. Mathias for critical readings of this manuscript.

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